



Survey of Sub-Micrometre Powder Size Measurement for Ceramic and Cermet Powders

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ABSTRACT

This report is the result of a survey carried out to discover the measurement needs for sub-micrometre sizing in the ceramic and cermet industry.

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Approved on behalf of Chief Executive, NPL,
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INTRODUCTION

As part of a 3 year project on advanced materials, covering topics including ceramics and cermets, a survey was carried out under the microstructural characterisation task. This survey was intended to examine the extent of the use, and the problems encountered in measurement of, sub-micrometre sized ceramic and cermet powders. The premise behind this task was that although advances in powder preparation techniques have enabled the production of sub-micrometre powders, there is uncertainty whether this has been matched by similar progress in the field of particle size measurement.

RESPONSES

1 How would you best describe your organisation's involvement in powders?

Powder manufacturer/supplier	14	Powder end user	10
Powder sizing equipment supplier	5	Research	10

A total of 73 questionnaires were sent out to companies that were thought to have an interest in the measurement of sub micrometre ceramic or cermet powders. These included most of the UK based manufacturers and suppliers powder sizing equipment, several universities with large engineering ceramic research groups, as well as the major suppliers and users of hardmetal and ceramic powders. The list was restricted to users and manufacturers of technical ceramics as it was thought powders used in the refractory and tableware industry would not contain significant amounts of sub micrometre particles. In all 34 replies were received with a fairly even spread between manufacturers, end users and research industries. Not surprisingly the best percentage of replies to questionnaires sent came from the powder sizing equipment manufacturers with only two companies not responding.

2 What type of powders are you dealing with?

Oxide ceramics	21	Non oxide ceramics	14	Hardmetals	17
Other	2				

The figures for this question are somewhat misleading in that equipment manufacturers and research departments answered in all three categories. Perhaps the more revealing is that result for respondents dealing exclusively with one material was:- hardmetals 9, oxide ceramics 7, and non-oxide ceramics 2. This reflects that companies involved in hardmetal production tend to deal exclusively with those materials whereas the technical ceramic companies tend to be more diverse.

3 Do these powders contain a significant fraction of sub-micrometre particles?

Yes	30	No	4
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Most respondents thought that the powders contained significant amounts of sub-micrometre particles. Obviously this question is somewhat ambiguous for those that have different ranges of powders and the term "significant fraction" is open to interpretation. Nevertheless the clear conclusion from the answers is that most powders being used or investigated contain sub micrometre particles.

4 What methods do you use for powder sizing?

BET	11	Gravity Sedimentation	8	Centrifugal Sedimentation	4
SEM/TEM	12	Electric zone sensing	4	Laser Scattering	21
FischerSSS	8	Not applicable	4		

The responses showed a wide variety of measuring techniques are being used, mostly those that had been suggested on the questionnaire, although a number of respondents (all involved with hardmetal powders) used Fischer Sub Sieve sizing, a technique not thought to be useful for sub micrometre measurement. The laser based techniques seemed the most widely used, and there are a number of similar instruments on the market that use this

method. Many respondents had more than one technique available to them and although the number of SEM/TEMs in use for sizing was surprising all the users with an SEM also had another technique available suggesting that the SEM methods are used as a backup or check. The 'not applicable' responses came from some powder end users who do not have any measurement equipment who rely on the suppliers for size information but are nevertheless interested in the measurement issues.

5 What do you consider to be the lower resolution limit of your techniques?

> 1 μ m 3 1- 0.5 μ m 4 0.5- 0.1 μ m 12 < 0.1 μ m 10
Not applicable 5

This question was ambiguous for those that used more than one technique and 'depending on test method used' was a frequent proviso in the responses, and where two values were given the lower value has been counted. The purpose of this question was to find out whether the users thought they had techniques available for measuring sub micrometre. The question is open to some subjectivity as to whether to give the theoretical or manufacturers minimum value or a value based on experience with the equipment. However generally most users felt they could measure to below 0.5 micrometres.

6 If you use more than one method, is there good agreement between the tests?

Yes 6 No 11
Not applicable 13 Yes but... 4

The large number of not applicable responses came from users that only used one method of measurement and those that had no experimental equipment. Although there were six unequivocal yes responses most of the no responses added comments such as 'this is not unexpected', or 'hardly surprising' and there were also four guarded yes responses termed 'yes but ...'. The general comments were that results from different methods did not give direct numerical agreement but in a qualitative sense there was a correlation, i.e. powders can be ranked in size consistently by all the methods.

7 Is there correlation between your powder size measurement and the processability or final product performance?

Yes 25 No 1 Not Applicable 4 Don't Know 4

This question was intended to discover if the size measurement was giving useful information for QA purposes or research investigations. Apart from the not applicables, or don't knows where the respondents did not measure the final product, and one 'no' response from a manufacturer of powder with a particle size greater than 50 micrometers, most respondents felt happy that the size measurement was reflected in the final product performance.

8 What steps do you use to disperse powders and reduce agglomeration?

Milling 20 Ultrasonic Agitation 23 Chemical dispersant 23
Not applicable 2

With hindsight this question should have had 'during particle sizing' added on to the end, as it was apparent that some respondents thought that this question referred to dispersion during processing of the powders. The question was intended to find out whether the respondents had addressed the issue of agglomeration during measurement and since there were no 'none used' replies this suggests that there is a general awareness of the problems.

9 What powder characteristics are you measuring?

Mean Size 30 Size distribution 27 Morphology 12
Other 1

This question was intended to find out what level of information was needed, if mean particle size is sufficient or if the complete distribution is necessary. Although most

respondents measured mean size many users also needed the full size distribution. The morphology response was included for the SEM/TEM measurement methods but there were also respondents that felt using BET (a surface area measurement) gave information on surface area and thus morphology.

10 What basis are your results expressed in?

Number fraction	7	Volume or Mass distribution	30
Not applicable			

Most respondents indicated that the basis for the size was expressed in volume or mass terms and only two respondents used number fraction exclusively to express the results. The question was intended to highlight the difference between the counting techniques such as SEM/TEM and those methods that measure the mass or volume of a certain size particle. In theory these two forms are interchangeable using a simple transformation but great care must be taken when converting wide size distributions between mass and number fractions because of the r^3 dependence of the volume.

11 Do you use any standard written procedures for your particle size measurements?

No	6	Internal procedures	24	British Standards	2
ISO standards	2	NAMAS Accredited procedure	2		
Not applicable 7					

From the responses given there would seem to be a great deal of standards and guidelines available but very few cover the tests in entirety so most people tend to use in house written procedures. Two of the respondents are using NAMAS accredited procedures and one is awaiting accreditation. The NAMAS accreditation is probably only viable for the companies providing an external measurement service, but most alarming is the six who do not have any written procedures.

12 Do you use any standards/ reference materials to calibrate your particle sizing equipment?

Industry based standards	7	Standards internal to your organisation	18
Standards traceable to the National Measurement System	10	None used	4
Not applicable 3			

This question was intended to show the level of traceability of the equipment used and although most used some sort of standards to calibrate the equipment four said that standards were not used. Clearly if measurement is used purely for in-house quality assurance purposes then traceability is not necessary and only consistency of measurements is required, and although some instruments do not need to be recalibrated or adjusted it is always useful to have some form of 'standard' to carry out checks.

13 Do you think that traceability to the NMS of your measurements is important?

Yes	19	No	10	Don't Know	2	Not Applicable	3
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The issue of whether quality assurance type measurements need to be traceable was again raised here. Interestingly that although 19 respondents said that NMS traceability was important at present only 10 actually perform measurements with traceable standards to calibrate their instruments. The traceability of standards obviously depends on the measurement technique and the comments showed an awareness of the various traceability routes.

14 How repeatable are your measurements?

Always	12	Only for the same operator	2	Yes but	13	Not Applicable	3
Don't know 4							

The majority of responses stated that their measurements were repeatable, with the 'yes but' category generally agreeing but with provisos. If a measurement was not repeatable it could

usually be traced to operator or equipment error, and in line with responses to question 6, different test methods did not give repeatable measurements on the same powder.

15 Do your size measurements agree with those obtained from other organisations?

Agree 11 Disagree 6 Don't know 8 Yes but 6 Not applicable 3

The responses to this question were widely split, with eleven agreeing with other organisations and six agreeing with conditions such as 'mostly agree', or 'only if the same method is used'. Powder manufacturers ran into problems in correlating their powder sizes with their users' results, especially if different methods are used, and powder users wanted to be sure of consistent results if they used different suppliers.

16 Would you be prepared to participate in an interlaboratory exercise, perhaps involving measuring three powder samples?

Yes 24 No 5 Not applicable 3 No answer 2

This question assesses the respondents interest in the problem since it asks them to do some work, and with 24 positive replies it seems that there is considerable interest in carrying out the round robin exercise. Some comments pointed out similar exercises had already taken place, and it is hoped to get details of these so that there is no unnecessary repetition.

17 Would you be interested in receiving the results of this project?

Yes 33 No 1

All the participants responded positively to this question except for one manufacturer who dealt with powders larger than 50 micrometers.

18 Do you also encounter measurement problems when sizing powders above 1 micrometre?

Yes 9 No 21 Not applicable 4

This question was intended to discover if there were any problems in sizing powders above 1 micrometre, and the majority response was no, and the nine yes responses did not highlight similar problems. It would therefore seem that sizing powders above 1 micrometre is not a widespread problem.

General Comments

In the general comments area three participants expressed their general interest in the work and thought it was a 'good idea'.

CONCLUSIONS

From the response to the questionnaire it would seem that there are powders being used or at least investigated that contain a significant proportion of sub-micrometre particles. A wide variety of techniques are being used in an attempt to measure the size distribution, with the laser based instruments being the most popular. Most users believe that they have the equipment capable of measuring down to these size ranges and although few users stated categorically that they could not size below one micron, nobody said that they had no problems or that they disagreed with the premise behind this exercise. There was general consensus that using different test methods gave different results and did not think that this was surprising. Obviously some methods measure different physical properties, however there is no reason (apart from statistical significance) why for instance the SEM and the laser scattering methods should give different results. Again most users felt that the results they were getting from powder sizing experiments correlated with the final product performance suggesting that they are not unhappy with their measurements.

From the standards aspect most respondents used their own internal procedures to carry out the measurement, and the calibration standards are also often only internal. Although there

was some split as to whether calibration standards needed to be traceable for internal quality assurance purposes, most felt that the standards should be traceable. Looking at the need for a round robin exercise, although most users felt their own measurements were repeatable, they often disagreed with other organisations, particularly if different methods were used, and most respondents were willing to take part in the round robin.

ROUND ROBIN EXERCISE

In order not to burden the participants with too much work the round robin will consist of three powders. A narrow size distribution hardmetal powder, and two ceramic powders, a wide and a narrow size distribution all containing sub micrometre particles. Participants will be asked to measure the wide size distribution ceramic and at least one other by the method of their choice, and to return the results together with a reason for their choice of method. At the same time NPL will try to provide traceable measurements of the powders, probably by SEM/TEM methods.

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